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PECVD of undoped silicon glass film on 300mm wafers

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PECVD of UNDOPED SILICON GLASS FILM on 300mm WAFERS

A Master of Science in Engineering Thesis

Presented to

The Faculty of the College of Engineering San Jose State University

In Partial Fulfillment

of the Requirements for the Degree

Master of Science in Engineering

by

Tom Kamp

May 2001

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ABSTRACT

PECVD of UNDOPED SILICON GLASS FILM on 300mm WAFERS

By Tom Kamp

The conversion from 200mm to 300mm wafers introduces many new process challenges. Plasma enhanced chemical vapor deposition (PECVD) processes are no exception. The development and characterization of an Undoped Silicon Glass (USG) thin film was performed on 300mm wafers using a Mattson Technology R & D PECVD system. The PECVD chamber was designed specifically for 300mm wafers and contained improvements over the 200mm system. Process characterization was done covering parameters such as deposition rate, thickness and uniformity, refractive index, stress, step coverage, particles, and metallic contamination. It was found that this scaled up 300mm size dual plate diode type PECVD reactor is able to meet and exceed industry specifications. Over a hundred process conditions were tested resulting in a USG film that meets the industry standards for 300mm size wafers. This thesis is the analysis and documentation of those results.

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1.0 Introduction

The following thesis is on the development of a USG film using a Mattson Technology Inc. Aspen III PECVD reactor.¹ The objective of this project is to develop a USG film on 300mm sized silicon wafers which has equal or better qualities than the Aspen II 200mm films and meets the current industry 300mm specifications set by Selete Japan. The industry specifications for a PECVD USG film on 300mm wafers at .25 μ m dimensions is shown in Table 1.1 and on the Selete web site.² The 300mm Aspen III platform and process chamber are complete redesigns over the current Aspen II design. It was found that with improved chamber design and improved process recipes, the 300mm USG film had several parameters that exceeded previous 200mm specifications and new 300mm industry standards.

Table 1.1 Target industry specification for USG film on 300mm silicon wafers.² (Selete 1998)

Parameter	For .25 μ m dim.	
Deposition Rate	>5500 Å/min	
Uniformity	< 3.0% (range)	(49pt 3mm ex)
Refractive index (RI)	1.46 $\pm$ 0.01	@633nm
Stress	-100 to -200Mpa	Compressive
Wafer to wafer	< 3.0% (range)	Range uniformity
Metallic contamination	<1.0E11 atoms/cm ²	Na,Cr,Fe,Ni,Cu,Zn,
Particle, in film	<30	Size >.20 μ m
Step Coverage	>60%	Of conformal

2.0 Background on PECVD

2.1 Literature Review

Several articles and text publications were found each pertaining to different portions of this project. No review articles were found that cover the entire scope of this paper. For the purpose of this paper the experimental results were compared to several articles, books, and Aspen II 200mm PECVD data which have been referenced throughout the discussions. The review articles and texts referenced contain data to support and expand upon some of the findings for a typical USG film. Smith's text, chapter 9 Glow-Discharge Plasmas, gave the most comprehensive explanations for general plasma CVD reaction mechanisms.⁴ However certain observations of the plasma behavior were visually seen during the experiments and no supporting reference material could be found. In this case these explanations were generated during the writing of this paper.

2.2 USG in Integrated Circuits

USG or silicon dioxide, has many uses with in Integrated Circuits (IC's). USG can be used as an insulator between metal layers and polysilicon, between multi layers of metal interconnects, as diffusion and implant masks, and as a passivation layers.⁸ Figure 2.1 shows USG as an Intermetal Dielectric (IMD) in a simple MOS device. Some of today's logic devices can have over 5 layers of metal each requiring a IMD. The USG insulates the metals lines from each other and provides a foundation for the next layer.

Figure 2.1 USG as an Inter-Metal Dielectric (IMD) used in a simple MOS device.

2.3 Plasma CVD³

Plasma enhanced CVD processes have a much lower deposition temperature than thermal processes. This makes them suited as inter-metal dielectrics (IMD) where thermal CVD processes would melt the already patterned aluminum interconnects. The energy provided by the plasma creates activated molecular species with increased potential energies. This lowers the required thermal energy required to activate the reactant gas molecules and complete the desired chemical reactions. The reaction rate is dependent primarily on two reaction controlling mechanisms of CVD; surface reaction and mass transport. The slowest mechanism will determine the rate.

2.4 PECVD Kinetics⁴

By altering the recipe conditions (flow, pressure, power, etc..) the plasmas internal reaction mechanisms can be changed by any one or more of the following regions (a - g) shown in Figure 2.2. A comprehensive discussion on PECVD Kinetics can be found in Smith.⁴ The primary chemical activated event is electron impact

dissociation to produce free radicals as shown at (a) from Figure 2.2. Ionization also occurs but at a much lower rate because of the higher energy required. The free radicals may diffuse directly to the surface (b), or they may first react with other radicals or molecules (c) to produce gaseous precursor molecules which then diffuse to the surface (d). Some of the source gas reaches the surface with out activation (e) and some is pumped away directly (f). Surface reactions begin after the reactant gas species absorbs on the wafer surface. The surface reactions may be activated by electron and ion bombardment and by the substrate temperature, T_s . This can promote the molecules to react with other absorbed reactant molecules, migrate or diffuse on the surface, and/or nucleate to form the film (g). The key parameters for film growth are adequate deposition temperature for surface chemical reactions to take place and sufficient reactant species concentrations.

Figure 2.2 Plasma-CVD reaction steps.⁴

Mass transport limited or diffusion limited reactions occur when the reactant molecules (b, d) are slow to diffuse to the wafer surface and are the limiting factor for film growth. The reactive gas molecules must make it through the bulk plasma to the wafer surface before becoming non-active or pumped out. Typically processes of high

temperature and high pressure fall into this diffusion controlled regime. In addition, for PECVD processes, the generation of available radicals by electron impact (a) are needed to diffuse to the surface which can limit the mass transport mechanism.

Surface reaction limited processes have plenty of available reactants (mass transport) but are limited by insufficient surface energy. Typically processes of low temperature and low pressure fall into this surface controlled regime.

Compared to typical LPCVD and APCVD processes, the operating PECVD temperature for this process (400°C) is low. The operating pressure (2.1 torr) could be considered high compared to LPCVD which typically runs below 1.0 torr.

By analyzing the results in the proceeding sections and by the temperature and pressure ranges used, it appears that the developed USG film has both mass transport and surface reaction limitations.

2.5 Mattson Technology Aspen PECVD Series

The Aspen platforms are designed to handle and process multiple wafers increasing through-put while maintaining simplicity and low operating costs.⁵ The Mattson Technology Aspen II and III PECVD reactor's basic design follows the principles of a typical diode type plasma reactor.^{6,7} Figure 2.3 depicts a basic schematic of the Aspen III parallel plate diode reactor. The Aspen II reactor can process wafer sizes of 100mm to 200mm(4 to 8 inch) and the Aspen III platform can process 300mm(12 inch) wafers.

Figure 2.3 Mattson Technology Inc. PECVD Reactor basic schematic (1998).¹

Wafers are robotically loaded from the vacuum transfer chamber through a pneumatic slit door onto the heater block. This ensures that the process chambers are always at vacuum. In the center of each wafer pocket there are three lift pins (or a rotating spindle in the case of Aspen II) which handles the wafers between the heater block and vacuum transfer robot. The thick aluminum heater block uses a resistive element welded within to create excellent temperature uniformity. Thermocouple (TC) wafer tests on both systems support a $\pm 5^{\circ}\text{C}$ range uniformity at 400°C . The heater block surface is hard anodized to protect the aluminum from being damaged during process, maintain repeatability, and to keep it from getting fluorinated during plasma cleans. The target operating temperatures of the heater block are 300°C to 400°C . A programmable controller controls temperature.

Process gas enters the chamber through holes on the two powered showerhead electrodes (4 showerheads for the Aspen II reactor). Showerhead hole patterns were designed to optimize gas distribution over the wafer. All gases are controlled by sized MFC's before entering a mixing manifold. From the mixing manifold the gases are evenly distributed to the faceplates (showerheads).

RF power is supplied by two generators; 3KW 13.56MHz and 1KW 200KHz. The generators are controlled by analog signals and different power and frequency ratios are used to alter film parameters. RF power passes through a matching network followed by a ballaster unit to split the power between the two electrodes (showerheads). The Aspen II uses a four way RF splitter assembly to evenly distribute the RF to each electrode. The heater block is the designated cathode.

2.6 Mattson Technology Aspen III PECVD

The Aspen III Mattson reactor is sized to accept two 300mm wafers side by side. Careful attention was taken in the details of the chamber components improving all films processed through it. Processing two wafers at once will greatly improve the systems overall throughput over competing systems single wafer design. The Aspen II reactor can hold up to four wafers but needs to deposit the film in more than one step by rotating the wafers to different showerheads reducing through put compared to the Aspen III. The goal of the Aspen III dual wafer design is maintaining good thickness uniformity while depositing the full thickness in a single step rather than performing station averaging over multi depositions.

All materials used inside the chamber are resistant to plasma etching and reduce contaminate emissions. The previous Aspen II reactor contained inconel bolts and other metal parts that were expose to the plasma. The Aspen III reactor contains no steel or inconel fasteners.

All chamber and handler components are controlled by software coded in a Windows NT operating system. The Aspen II uses a QNX operating system which displays in real time and is very robust but lacks process parameter flexibility and complicates adding new features.

Chamber free volume (empty space) was reduced approximately by a factor of seven to ease chamber cleaning times and restrict particle accumulation. The smaller chamber interior surface area makes it possible to plasma clean (plasma etch) all deposited films faster with no residual spots which could be a source of particles. The Aspen II reactor had much open space under and around the heater block whereas the Aspen III PECVD uses ceramic parts to fill all of the non process critical spaces.

3.0 Experimental Design

3.1 Experimental Reasoning

Several parameters must be understood to completely characterize the USG film. Parameters to be researched include thickness uniformity, deposition rate, refractive index, film stress, step coverage, particles and metallic contamination. The ultimate goal is to qualify the Aspen III PECVD system by developing a repeatable USG process that meets the current industry 300mm specifications set by Selete Japan.²

3.2 Experimental Design

Using the software environment a recipe page was generated to alter the reaction by varying components of the process; deposition time, pressure, high frequency power, low frequency power, temperature, and individual gas flows (SiH_4 , N_2O , N_2). A blank recipe page is shown in Table 3.1. The desired reaction is $\text{SiH}_4 + 2\text{N}_2\text{O} \rightarrow \text{SiO}_2 + 2\text{N}_2 + 2\text{H}_2$. The diluent gas is N_2 . The Aspen III windows NT operating environment gives more flexibility in recipe building than does the previous QNX environment. The Aspen II system uses a QNX system in which the software was coded with one step for process with purges and silane dumps as separate features.

Table 3.1 Recipe page for process parameter configuration.

Step #	1	2	3	4	5
Time (sec.)	0	0	0	0	0
SiH ₄ dump(sec)	0	0	0	0	0
Pressure (mtorr)	0	0	0	0	0
H. Freq. (W)	0	0	0	0	0
L. Freq. (W)	0	0	0	0	0
N ₂ (sccm)	0	0	0	0	0
N ₂ O (sccm)	0	0	0	0	0
SiH ₄ (sccm)	0	0	0	0	0
Temp. (°C)	0	0	0	0	0

All of the experiments were conducted by making slight changes to one parameter at a time and plotting the resulting trends. For example, five wafers were run with each one processed with a different pressure. All of the film parameters (i.e. rate, uniformity etc..) were plotted and the pressure which favored the most desirable film parameter was used to characterize the next process component. Now gas flow could be varied while maintaining the favorable pressure and so on. Later when the process has been shifted to a more desirable regime, all the same parameter trends were processed again starting from the improved baseline recipe. All process parameters were run several times with different recipes. Almost a hundred plots were made until a recipe was reached which met all of the required industry film specifications.

Although the developed process is within the specifications, it is not optimized. To optimize the process would require a complete surface response Design of Experiments (DOE).

3.3 Metrology

Thickness uniformity was performed using a Tencor 1250 and Nanospec 8300x film thickness measuring equipment. All 300mm wafers were measured with a 49 point 3mm edge exclusion stage configuration. Data collected was the mean thickness, percent standard deviation, and percent range uniformity.

$$\text{Percent range uniformity} = 100 * (\text{max-min}) / (2 * \text{average})$$

Details on all of the included thin film metrology techniques can be found in Sherman.³

Deposition rate was calculated using the wafer thickness mean from each wafer and dividing it by the process time. The process time is the total time contained in the plasma deposition step in the recipe shown in Table 4.1. Wafer heat soak, gas stabilization, post RF, and purge step times were not included in the rate calculation since no deposition occurs there.

Refractive index was measured using a Metricon prism coupler and an automated KLA-Tencor 1250 system. The Metricon was used to measure a single point and the KLA-Tencor 1250 measured the refractive index using 49 point with a 3mm edge exclusion stage.

Wafer surface particles were measured using a Tencor SP-1 surface scan system & Topcon WM-3000 surface scan system. All wafers were pre-measured counting all pre-existing particles contained on the wafer surface. All particles above $>.16\mu\text{m}$ were mapped for wafer transfer and particles $>.20\mu\text{m}$ for in-film particle maps. A reference wafer was used in all cases to judge the operating environment.

Film Stress was measured using a FSM 128 surface deflection system. All wafers were pre-scanned before processing to factor in any bare wafer deflection.

4.0 Results & Discussion

4.1 Overview

All results shown were generated from slight recipe changes around the developed baseline to explore the process window. The baseline (center) process was arrived at by exploring the process capabilities of the reactor until all parameter were in specification. Much of the exploratory data is in a different process regime and is not included. The applicable raw data is included in the appendix. The process recipe step 3 in Table 4.1 depicts the developed process. The other recipe steps are used for gas stabilization, wafer heat soaking, and post plasma for flushing the remaining silane gas. The USG (oxide) soak time used was 10 seconds. A 10 second soak time is short enough to not decrease overall wafer throughput and long enough so the 5000Å USG film properties are not compromised. The soak time data can be reviewed in the appendix. This gives the wafer enough time to ramp up to a reasonable process temperature. The gas stabilization is performed during the soak step and helps heat up the wafer by convection heating.

Table 4.1 Developed 5000Å USG process recipe.

Recipe	Pump out	Gas Stblz & soak	Deposit	Post RF	Purge
Step #			3		
Time (sec.)			59		
SiH ₄ dump(sec)			0		
Pressure (mtorr)			2100		
H. Freq. (W)			581		
L. Freq. (W)			194		
N ₂ (sccm)			1580		
N ₂ O (sccm)			8160		
SiH ₄ (sccm)			260		
Temp. (°C)			400		

The developed process recipes results can be seen in Table 4.2. Film thickness uniformity and Refractive Index (RI) distribution maps are in Figures 4.1 & 4.2 below.

Table 4.2 Developed USG process results.

Parameter	Specification	Results
Deposition Rate	>5500 Å/min	6150Å/min
Uniformity	< 3.0% (range)	< 3.0% (range)
Refractive index (RI)	1.46 ± 0.01	1.465 ± 0.005
Stress	-100 to -200Mpa	-100 to -200Mpa
Wafer to wafer	< 3.0% (range)	< 1.5% (range)
Metallic contamination	<1.0E11 atoms/cm ²	Yes
Particle, in film	<30	Yes, Process only
Step Coverage	>60%	≈ 60%

Figure 4.1 3D thickness uniformity map of the developed USG film.

The 3D thickness map in Figure 4.1 shows how the film is thicker on the edges. The blue line circling the interior of the surface represents the average thickness at 5112Å. The 2D refractive index map in Figure 4.2 is of the same wafer. The map shows how the refractive index is lower at the wafer's edge. The thick red line represents the average refractive index for this wafer at 1.465.

Figure 4.2 2D contour map of refractive index distribution.

Each process parameter varied affects each response differently. The following trend map, Figure 4.3, and effect pareto Figures 4.4 – 4.7 show how each factor effects the responses; deposition rate, refractive index, uniformity, and stress. The trend map shows the effects of each factor being increased and decreased around the center value from the developed process. The following trend map also suggests that the developed process, even though within spec, can be optimized by a DOE. The map shows how some factors, total flow, pressure, total power, %low freq. power, and %SiH₄, have stronger trends than others, N₂ flow and temperature. For example, it appears that uniformity could be improved and deposition rate slightly increased if % low freq. and %

SiH₄ were simultaneously increased from center. Performing a DOE could find these regimes that don't show up on the one dimensional trend maps.

Figure 4.3 Trend map for all factors varied with applicable reference numbers.

The effects pareto Figures have each factors relative strength plotted against an individual response. For example in Figure 4.4, % silane shows the strongest influence for increasing the deposition rate while % low frequency appears to decrease the deposition rate.

Figure 4.4 Effects Pareto for deposition rate.

Figure 4.5 Effects Pareto for refractive index.

Figure 4.6 Effects Pareto for thickness uniformity.

Figure 4.7 Effects Pareto for film stress.

4.2 Thickness uniformity at the wafers edge

In most process conditions tested, the thickness uniformity maps show a wafer edge thick film. This means that the wafer thickness is greater near the wafer's edge. This effect can be seen on the Aspen II reactor however, most of the thick edges are not

reported by data because of that generations 6mm edge exclusion uniformity measurements. Edge exclusion is the distance from the edge of the wafer that is excluded from the thickness measurements. All 300mm wafer specifications use a 3mm edge exclusion measurement which is close enough to the edge to measure this phenomena. Still up for discussion is what is really happening at the wafer's edge; no applicable references were found. The thin films text by Smith describes plasma coupling effects with surfaces and sheaths but no explanation on wafer edge effects.⁴

Possible explanations could be narrowed down to some ion energy and/or direction changes through the sheath as it steps over the rounded wafer's edge. This adds curvature to the sheath bringing more ions towards the edge altering the deposition. Thus making ion surface activation an important part of the film growth. The following Figure 4.8 shows a cross section of the rounded wafer edge extending in 0.35mm(350 μ m); its potential thickness uniformity effects may extend further.

Figure 4.8 SEM cross-section of 300mm wafer edge.

Another explanation could come from the diffusion mechanism discussed earlier where the edge may be more likely to have a higher concentration of radicals and

precursors. The center of the wafer allows for equal distribution of reactants in all directions. The edge has additional flux of species from the side. A moderate sticking coefficient (as discussed in section 4.12 step coverage) may cause this excess flux of species to increase the build up of film at the edge.

Experiments were performed with smaller 200mm wafers in the same reactor under the same conditions and the edge thickness results were comparable. This showed that this effect was not dependent on position in the reactor but followed the position of the silicon wafer edge. Another experiment was performed by placing silicon wafer pieces around (touching) a 200mm wafer thus partially eliminating the edge and performing the same deposition. The thickness profile was much smoother where the wafer pieces touched the test wafer. Pressure and percent low frequency power changes, discussed later, can also slightly improve this edge effect. More investigative work will have to be done to reduce the edge effect for future, most likely lower uniformity, specifications.

4.3 Total flow variable

The total flow includes all gases entering the chamber during process. Each gas was varied by it's respective percentage of the total flow. Figure 4.9 shows four plots of how the deposition rate, thickness uniformity, refractive index, and film stress are effected by varying the total gas flow.

Figure 4.9 Total gas flow effects.

It was found that 10,000sccm of total gas flow gave the best results. The most sensitive parameters effected by the overall flow were within wafer thickness uniformity and deposition rate specifications. This trend also applies for the Aspen II system where the optimum total flow is 6,595sccm. At 10,000sccm the gas shower heads maintain enough back pressure relative to the chamber pressure to let the gas uniformly exit all of the inlet holes perforated in the electrode. Increasing the flow further tends to amplify the conductance differences of each individual hole or imperfections in the shower head.⁸ This amplification of the shower heads machined tolerances begins to mirror itself onto the wafer creating minor different thickness' along the substrate. This problem should disappear as the manufacturing process is matured for later shower head revisions.

It is apparent from the data that rate is proportional to the total gas flow, Figure 4.9. More gas entering the chamber introduces more reactive species into the plasma thus

increasing the deposition rate. When the total gas flow reaches 10,500sccm the plasma becomes saturated and the rate no longer increases, Figure 4.9. This could be related to the residence time of the reactants.⁹ Residence time is the mean time a gas or molecule remains in the chamber before being pumped away. At higher flows the gas must be pumped faster to maintain the same pressure, reducing the residence time. The increased flow is flattening the reaction rate because the excess gases now become a diluent and evacuated from the chamber without reacting. When the rate flattens out the reaction appears to become mass transport limited. More reactants are being supplied to the plasma but the same amount are absorbing to the surface.

4.4 Pressure variable

The chamber pressure was varied incrementally within a pre-observed boundary in which a stable glow discharge could be sustained. Maintaining plasma stability is important for uniform distribution of reactive species in the glow discharge which normally results in better film thickness and compositional uniformity.⁶ Pressure changes provided enhanced control over the plasma focus. The focus can be described as the sharpness of the plasma column visually seen between the upper (shower head) and lower (wafer on heater) electrodes. This focusing effect can be seen through the chamber view ports, Figure 4.10, and is explained as the decreasing plasma reach over the grounded electrode.⁴ Another way to describe the observed plasma focusing effects at a molecular level is by describing the atomic and molecular excitations.⁹ The light emitted is in the form of photon energy when an excited electron returns to its ground state (or any lower

state). At higher pressures there are more frequent atomic and molecular collisions. The collisions enhance the transition of electrons from the excited states to lower states. Therefore an excited species will travel less distance at higher pressure before being relaxed back to its ground state. This means that at lower pressure the light (photon energy) will be more spread out over the reactor because of the increase excited electron state lifetimes, Figure 4.10. The developed process was run at 2100mtorr with a deposition rate of 6200 Å/min. Aspen II 200mm Oxide process runs at 2650mtorr with a deposition rate of 4100 Å/min.

Figure 4.10 Higher pressure plasma focus (A), Lower pressure plasma (B).

Figure 4.11 Pressure variable effects.

Higher pressures created a more focused and brighter plasma over the wafer which affected the deposition rate, increased the thickness uniformity, and decreased the refractive index, Figure 4.11. The brighter, more focused plasma is an indication of more excited species which contributes to more ionization.⁹ The deposition rate on the wafer is reduced at pressures below 2100mtorr because some of the deposition is now also occurring elsewhere in the chamber. This is due to the longer ion mean free paths and resulting less collisions producing less ionized reactants to the surface. The deposition rate peaks at 2100mtorr and then begins to decrease with increasing pressure, Figure 4.11. No supporting USG trends were found but similar silicon nitride results indicated that the deposition rate can increase and/or decrease depending on pressure.¹⁰ Increasing the pressure increases the residence time of the molecules which promotes more recombination of the ionized reactants which now have a much shorter mean free path.⁹

This creates a diffusion limitation within the bulk plasma reducing the amount of useful reactants.

Lower pressure gave better thickness uniformity. At higher pressures the plasma visually appeared non uniform and the wafer film thickness became thicker around the wafer edges thus enhancing the edge effect discussed earlier. Pressure changes also affect the gas shower head back pressure, thus slightly simulating the process conditions changed by total flow variations. High pressures reduce the showerhead back pressure resulting in a less uniform gas flow from all of the holes in the electrode (showerhead). Lower pressures increased the showerhead back pressure forcing the gas out evenly giving improved uniformity.

4.5 Total high & low frequency power variable

Both the high and low frequencies wattage output was varied while maintaining their relative percentages. Refractive index and deposition rate and were the most heavily influenced by changes in the total RF power, Figure 4.12. Refractive index decreased with increasing power.¹¹ This is an indication that the film becomes less silicon rich and possibly contains a higher percentage of hydrogen.

As the total wattage applied to the electrodes was increased, the deposition rate increased. More power means more energy is being supplied to the reaction which increases the disassociation and ionization freeing up more activated species to the substrate, thus increasing deposition rate. This supports the generation of radicals being a rate limiting step. The increase in deposition rate with increasing RF power is supported

by many other PECVD publications and previous Aspen II CVD data and another review articles.^{3,10,11}

Figure 4.12 Total RF power effects.

Unfortunately increased deposition rate by power affects the uniformity. After about 775W of total power, the percent range thickness uniformity begins to increase, Figure 4.12. The plasma focus visually becomes less uniform which may be related to non uniform ionization rates within the plasma translating to an uneven film growth. This assumes areas of higher photon energy emission also have different ionization rates of the relative species. Optimizing the rate and still maintaining acceptable uniformity was achieved at 775W of power.

Using the wafers size and total power differences between the Aspen II and Aspen III systems, the power per mm^2 can be calculated, see the following Table 4.3.

The wafer sizes will be used since the electrode diameters for both 200 and 300mm reactors is only slightly larger than the wafer diameters. The Aspen III reactor uses less power per area of silicon wafer than does the Aspen II optimized process implying a more efficient power delivery. This could possibly be attributed to the Aspen II chamber volume being approximately seven times the Aspen III changing some of the reactant mechanisms like residence times.

Table 4.3 Power per mm² calculation using wafer size.

Power work sheet			
	Aspen II	Aspen III	
Total power	900	775	watts
Wafer dia.	200	300	mm
# of Heads	4	2	per chamber
Power per head	225	387.5	watts
Area per wafer (head)	31410	70672.5	mm ²
Power per mm²	0.0072	0.0055	watts/mm²

4.6 Percent low frequency power variable

The percent of low frequency was varied from a total wattage output between both frequencies. The results for each response are plotted in Figure 4.13. The most unique contributions from low frequency are its ability to alter the film stress,^{3,4} decrease the deposition rate and improve the thickness uniformity.

Figure 4.13 Percent low frequency effects.

Increasing the low frequency power creates higher ion energy which increases the physical bombardment on the film surface.⁴ Stress is induced into the film by the bombardment (ion-peening) which drives the film more compressive. It was shown that increasing the percent low frequency increased the compressive stress of the film, Figure 4.13. This is supported by other research.^{3,4} Aspen II 200mm silicon nitride process's also show this as a strong trend. The Aspen II USG process doesn't use low frequency power because of uniformity problems but when introduced for experimental reasons the stress also becomes more compressive or negatively stressed. Figures 4.14 - 4.16 show 3D maps of different compressive films stresses with 12.5%, 25%, and 37%LF powers. The lower surfaces of the 3D maps indicate lower or more compressive stresses.

Figure 4.14 Stress profile showing -107Mpa Compressive (view angled 40°). Low frequency power was 12.5% of the total RF power.

Figure 4.15 Stress profile showing -170Mpa Compressive (view angled 20°). Low frequency power was 25% of the total RF power.

Figure 4.16 Stress profile showing -210Mpa Compressive (view angled 30°). Low frequency power was 37% of the total RF power.

More low frequency power seemed to decrease the edge effect lowering the thickness uniformity. This may be attributed to the change in ion energies at the wafers edge.

4.7 Percent silane to nitrous oxide variable

Since silane and nitrous oxide are the two reactants. Their respective percentages were varied while maintaining the same total flow, Figure 4.17 Increasing the silane ratio drastically increased the deposition rate and refractive index. The refractive index increases because the film becomes more silicon rich.

Figure 4.17 % Silane effects

The refractive index is related to the atomic percentage of silicon contained in the oxide.¹¹ The final recipe contained 3.2% SiH₄ which differed from the Aspen II CVD

which was 4.0%. The percentage differences are most likely attributed to different powers, pressures, total gas flows and reactor design.

The deposition rate increased as more percent silicon was introduced. This is supported by other researchers.¹³ Silicon is the backbone for the SiO-SiO₂ film. Since the reactivity between silane and oxygen is very high, increased silane concentration will promote more reactant species available to the surface.⁶

4.8 Hydrogen content

Fourier Transform Infrared (FTIR) using a BIO-RAD system was done to check the films IR spectra components. SiO-SiO₂ peaks were strong with moisture content (H-O compounds) and Si-H peaks low. No data was extracted from these plots; only a visual confirmation was done. Typical plasma oxide spectra have hydrogen concentrations reported to be 5 – 10%.¹⁴ Hydrogen compounds exist in the form of Si-H, Si-O-H, and H-O-H. Electrical measurements show that plasma oxide films with minimal O-H bonds generally have better MOS electrical properties.⁶

4.9 Total nitrogen flow variable

The total nitrogen flow was varied while at the same time altering the total overall gas flow. Increasing the N₂ flow dilutes the gas mixture slightly decreasing the deposition rate, Figure 4.18. Nitrogen for the most part is not a reacting constituent in the film chemistry. It was found that 1,582sccm of N₂ gave the best uniformity results. The Aspen II N₂ flow trends are very similar optimized at 1,400sccm

Figure 4.18 Total N₂ flow effects.

Overdiluting the mixture with N₂ can worsen the thickness uniformity. When the non-reacting gas species flow becomes too high, the pump begins to control the thickness uniformity. The reactants closer to the pump port get evacuated with the N₂ diluent before reaching the substrate. Figure 4.19 below shows how 2,600sccm of N₂ affects the thickness uniformity. The thickness on the pump port side is lower.

Figure 4.19 Pumping effect on the thickness uniformity with 2600sccm of N₂.

4.10 Temperature variable

Temperature was varied while keeping all other recipe parameters constant. Higher temperature gave the most desirable film results. The temperature matrix was limited to a maximum temperature of 400°C because the reactor was not designed to run repeatability at temperatures well above 400°C. Also 400°C is an industry accepted temperature for PECVD reactors as well as what the Aspen II uses. The higher temperature slightly increased the deposition rate, Figure 4.20. This trend is supported by earlier papers.^{13,15} The small rate increase suggests that the surface reaction is not strongly influenced by temperature.

Also the film stress decreased as the temperature increased. This result is found in other research.¹³

Figure 4.20 Temperature effects.

4.11 Repeatability

In order for a process to be usable in a production fab it must be repeatable. Fifty-two wafers were run in order to check for process drifts, Figures 4.21 – 4.25. Two wafers were processed in the chamber at the same time because of the chambers side by side process capability. This means that for every deposition step, two wafers were simultaneously deposited on. Both the left wafer and right wafer film parameters were plotted to check any deviations.

Figure 4.21 Wafer thickness over 52 wafer run both left & right sides.

Figure 4.22 % Range uniformity over 52 wafer run both left & right sides.

Figure 4.23 Refractive index over 52 wafer run both left & right sides.

Figure 4.24 Deposition rate over 52 wafer run both left & right sides.

Figure 4.25 Stress variation over 52 wafer run both left & right sides.

The over-all run thickness range was 0.75%, Figure 4.21 (Also rate Figure 4.24), while all wafers had range uniformity's less than 3.0%, Figure 4.22. Refractive index showed a range uniformity of 0.02% thus proving that the process is repeatable. Figure 4.23. The stress values bounced around but generally stayed within the -100 to -200 Mpa specification, Figure 4.25. The stress measuring system (FSM 128) was also undergoing qualification tests so the repeatability wafers had to be measured over a couple of days per system availability. It's uncertain whether this contributed to the large stress range over the repeatability run and previous trend experiments.

4.12 Step Coverage

After a stable film was developed that met the specifications, the step coverage was checked. Step coverage was calculated by the aspect ratio of the side wall thickness and structure top thickness. The SEM photos taken show the USG film as deposited on structure wafers with an aspect ratio of 2:1 and 3:1, Figures 4.26 – 4.27

Figure 4.26 SEM cross section of USG on metal structure with aspect ratio of 2:1.

Figure 4.27 SEM cross section of USG on metal structure with aspect ratio of 3:1.

Figure 4.27 shows how the film forms a keyhole structure but is still >60% conformal before closing off the trench.

Silane based plasma oxides show good step coverage but are not typically 100% conformal especially for higher aspect ratio features. The film step coverage mechanisms and their results agree with Adams¹⁴ where silane plasma oxide films are found to also not be 100% conformal.

The keyhole or void forms because the reactants do not completely diffuse or migrate along the surfaces easily to the trench side walls because they have an intermediate to high sticking coefficient, S_c . Also, deposition carried by ions is perpendicular to the plasma and therefore deposits on the top surface and on the trench bottoms shown by region 1 of the Figure 4.28.⁴ The SEM results show that the step coverage mechanisms are driven partly by surface diffusion, region 3, and

ion activated deposition, region 1. Higher aspect ratios tend to keyhole easier as in Figure 4.27. In the case of $\text{SiH}_4 + \text{N}_2\text{O}$ based plasma deposited films, the reactants have an intermediate sticking coefficient.¹⁴ Species of low S_c , region 4, tend to be more conformal.

Figure 4.28 Hypothetical thickness profile of a deposited film into the test structure cavity. Deposition into numbered regions is mainly due to (1) ion activation, (2) neutral species of high sticking coefficient, S_c , (3) surface diffusion, and (4) low- S_c species. Smith.⁴

4.13 Particles

Particles have proven to be one of the toughest problems to overcome in plasma CVD reactors. Several precautions are taken to prevent particles from gathering on wafers during transfer and deposition. It is customary to pre-coat the chamber before running and then perform a plasma clean after some pre-determined thickness. In the case of this process, a $1\mu\text{m}$ oxide pre-coat was deposited in the chamber and a plasma clean was run after $10\mu\text{m}$ of deposition. Pre-coating the

chamber also increases the run to run repeatability and lowers first wafer effects by establishing a known chamber condition.

Special attention was paid to the recipe layout for particle reduction. The plasma was ignited 1 sec before the SiH_4 entered the chamber. At the end of the deposition step the SiH_4 was turned off 1 second before the RF turns off, labeled as post RF from Table 4.1. These special recipe configurations are to prevent any SiH_4 gas from being in the chamber without a plasma on. During the deposition process, homogeneous nucleation can occur where particles are formed in the plasma eventually becoming incorporated into the film. The nucleation rate is determined by the second order gas phase reaction between the film precursors.⁴ Increased pressure and RF power can enhance this nucleation rate. Gas phase nucleation typically shows up in the form of small particles $<0.8\mu\text{m}$.

Most of the particle problems encountered were of medium to large size. This was mainly attributed to other unrelated hardware tests that were simultaneously running on the system; slit valve tests, robot alignments, etc..

4.14 Metal Contamination

Metals can poison an IC by getting into the deposited film during processing or transfer. First wafers were run into the chamber with N_2 flow and back out. This is used as the reference wafer to baseline wafer transfer metal contaminates. The metals of interest are Na, Cr, Fe, Ni, Cu, Zn, Mn, and Al. Aluminum was not measured by this Transmissive X-ray Fluorescence (TXRF) technique. The reference wafer

showed no metal contamination above the specification 1.0×10^{11} atoms/cm², Figure 4.29

Figure 4.29 TXRF of reference wafers (no process).²

Next, contamination test wafers were run through a complete deposition cycle and measured, Figure 4.30. Traces of iron, nickel, and manganese showed up but were still within the specification. The plasma ion bombardment most likely sputtered these contaminants loose from somewhere in the chamber, possibly from the inconel bolts under the heater block.

元素	峰位 (keV)	峰面积 (cps)	含量 (wt%)	B.G. (cps)	σ (cps)
V - Lb1	9.67	1414.6380			
Si - Ka	1.74	1223.7880	133443.00	0.9855	1.439
S - Ka	2.31	2.8248	108.87	0.7783	0.074
K - Ka	3.31	0.0000	ND	0.5184	0.029
Ca - Ka	3.69	0.3284	3.01	0.4882	0.037
Ti - Ka	4.51	0.0000	ND	0.3816	0.025
Cr - Ka	5.41	0.0000	ND	0.3453	0.024
Mn - Ka	5.89	0.0818	0.14	0.3876	0.027
Fe - Ka	6.40	0.5879	1.02	0.3834	0.040
Co - Ka	6.93	0.0000	ND	0.3728	0.023
Ni - Ka	7.47	0.1214	0.15	0.4898	0.032
Cu - Ka	8.04	0.0000	ND	0.6520	0.033
Zn - Ka	8.63	0.0000	ND	1.1434	0.044
Ce - La	4.84	0.0000		0.2896	0.022

Figure 4.30 TXRF of processed wafer.²

4.15 Electrical testing

The final proving ground for the newly developed oxide is for it to be successfully integrated into a device. Device wafers were processed through the system to deposit the oxide inter-metal dielectric. The wafers were then sent through the rest of the process flow completing the device. After completion (weeks later) the 64M DRAM devices worked therefor qualifying the USG process. Details of this test are proprietary to the device fab at which they were run.²

5.0 Conclusion

Using a conventional dual plate diode type PECVD system it is possible to deposit USG films on 300mm sized silicon wafers that was proven to be a useful industry dielectric. The thickness uniformity, deposition rate, refractive index, stress, step coverage, metal contamination and particle counts meet the 300mm industry specifications of the time and exceed the performance of previous Aspen II 200mm USG films. The deposition rate exceeded the 5500Å/min target reaching 6150Å/min and the thickness uniformity was well below the 3.0% range specification. It was also discovered through data analysis that both mass transport and surface reaction mechanisms are factors during the USG deposition.

Even though the developed film meets all specifications, future work can be done to optimize it. A complete surface response could be done using DOE techniques. Also the thickness uniformity differences at the wafers edge needs further investigation through chamber hardware experiments.

Plasma Enhanced Chemical Vapor Deposition Glossary

Activation Energy	The minimum energy required to break the molecular bonds of reactants and to permit formation of new molecular bonds of products.
Capacitance	A measure of the ability of a material to store electric charge.
Compound	A composition of different elements that are chemically combined (molecules) and has different properties than the elements that make up the compound.
Compressive stress	The force parallel to the wafer surface per unit area that causes a deposited or growth film to contract or shrink.
Conformal	The conformality of a film refers to the degree to which a film maintains the same thickness and exactly follows the contours of the surface topography. Step coverage refers to the conformality over a step.
CVD	Chemical vapor deposition
DOE	Design of experiments
Dielectric films	An electrically non-conducting insulating material used to provide electrical isolation and physical protection of semiconductor circuits.
Edge exclusion	The distance from the edge of the wafer that is excluded from measurement. Usually configurable on the Metrology tool.
FTIR	Fourier Transform Infrared
Gas Phase Nucleation	The nucleation of film material in the gas phase instead of on the wafer surface.
Insulating films	Materials that have high opposition to the flow of electric current.

Interlevel Dielectric	An insulating material used to provide electrical isolation between conducting layers and planarization of surfaces of semiconductor circuits. (ILD)
Intermetal Dielectric	An insulating material used to provide electrical isolation between metal lines in the semiconductor circuit. (IMD)
Ionize	To separate a charge into ions.
IR	Infrared
LPCVD	Low pressure chemical vapor deposition which uses reactant gases at low pressure and at high temperatures.
MFC	Mass Flow Controller
Passivation Layer	The final layer of dielectric material used to protect the semiconductor circuit from moisture, contamination and physical damage.
PECVD	Plasma Enhanced Chemical Vapor Deposition
Photon	Energy released in the form of light
Radicals	An atom or collection of atoms which is electrically neutral but which also exists in a state of incomplete chemical bonding making it very reactive.
Reactants	The substances that react in a chemical reaction.
Refractive Index	The ratio of the speed of light through a material relative to the speed of light through a vacuum. The refractive index gives an approximate guide to the film's composition.
RF	Radio Frequency
Sheath	The positively charged layer where quasineutral plasmas are joined to surfaces.
Soak Time	Time in the recipe to allow the wafer to reach process temperature

TEOS	Tetraethylorthosilane
TXRF	Transmissive X-ray Fluorescence
USG	Undoped silicon glass or Silane-oxide; SiO or SiO ₂ .

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Appendix

A.1 Heat soak data

Tot. flow sccm	Pressure torr	N2 flow sccm	%LF %	Power watts	Temp C	%Silane %	Soak seconds	Rate A/min	RI	Unif. %
10000	2.10	1580	25.00	775	400	3.2	35	6194	1.4633	2.06
10000	2.10	1580	25.00	775	400	3.2	25	6176	1.4632	1.86
10000	2.10	1580	25.00	775	400	3.2	10	6136	1.4631	2.04
10000	2.10	1580	25.00	775	400	3.2	5	6089	1.4629	2.20

A.2 Raw Data (of included plots only)

Tot. flow sccm	Pressure torr	N2 flow sccm	%LF %	Power watts	Temp C	%Silane %	Rate A/min	RI	Unif. %	Stress Mpa
9000	2.10	1580	25.00	775	400	3.2	5576	1.4631	3.29	-121
9500	2.10	1580	25.00	775	400	3.2	6001	1.4629	2.16	-100
10000	2.10	1580	25.00	775	400	3.2	6183	1.4629	1.9	-95
10500	2.10	1580	25.00	775	400	3.2	6176	1.4634	2.05	-90
10000	1.90	1580	25.00	775	400	3.2	6098	1.4642	1.83	-50
10000	2.10	1580	25.00	775	400	3.2	6136	1.4634	1.42	-5
10000	2.30	1580	25.00	775	400	3.2	6059	1.4625	1.67	-54
10000	2.50	1580	25.00	775	400	3.2	5896	1.4617	2.62	-43
10000	2.10	1000	25.00	775	400	3.2	6185	1.4634	1.8	-132
10000	2.10	1580	25.00	775	400	3.2	6176	1.4633	1.76	-138
10000	2.10	2000	25.00	775	400	3.2	6229	1.4634	2.31	-149
10000	2.10	1580	12.50	775	400	3.2	6476	1.4626	4.18	-18
10000	2.10	1580	25.00	775	400	3.2	6169	1.4634	1.68	-82
10000	2.10	1580	37.50	775	400	3.2	5900	1.4634	2.17	-160
10000	2.10	1580	25.00	625	400	3.2	5684	1.4652	1.42	30
10000	2.10	1580	25.00	775	400	3.2	6153	1.4635	1.42	-12
10000	2.10	1580	25.00	925	400	3.2	6369	1.4621	2.22	-60
10000	2.10	1580	25.00	775	350	3.2	5971	1.4631	2.12	-58
10000	2.10	1580	25.00	775	375	3.2	6134	1.4629	2.05	-140
10000	2.10	1580	25.00	775	400	3.2	6181	1.4634	2.02	-159
10000	2.10	1580	25.00	775	400	2.9	5749	1.4622	2.21	-57
10000	2.10	1580	25.00	775	400	3.2	6168	1.4634	1.59	-41
10000	2.10	1580	25.00	775	400	3.5	6573	1.4638	1.65	8